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**PCT**

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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/US96/13827 <b>(22) International Filing Date:</b> 29 August 1996 (29.08.96) <b>(30) Priority Data:</b> 08/538,487 3 October 1995 (03.10.95) US <b>(71) Applicant:</b> ATLANTIC RESEARCH CORPORATION [US/US]; 5945 Wellington Road, Gainesville, VA 22065 (US). <b>(72) Inventors:</b> SCHEFFEE, Robert, S.; 9450 Jenerio Court, Lorton, VA 22079 (US). LARIMER, Merlin, W.; 1418 Virginia Avenue, Front Royal, VA 22630 (US). <b>(74) Agents:</b> PRESTA, Frank, P. et al.; Lowe, Price, LeBlanc & Becker, Suite 300, 99 Canal Center Plaza, Alexandria, VA 22314 (US).		<b>(81) Designated States:</b> AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, <u>FR</u> , GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> QUICK CURE HYDROXYL-TERMINATED BINDER SYSTEM FOR GAS-GENERATING COMPOSITIONS		
<b>(57) Abstract</b> <p>A gas-generating composition for inflating inflation devices such as air bags, life rafts, slide chutes, and the like which comprises an oxidizer component and a hydroxyl-terminated polybutadiene binder component which does not produce hazardous compounds upon combustion. The composition further includes a curing agent and a cure catalyst so that it can be molded and quick-cured. Molded articles can be formed by injection molding or extrusion.</p>		

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QUICK CURE HYDROXYL-TERMINATED  
BINDER SYSTEM FOR GAS-GENERATING COMPOSITIONS

Technical Field

The present invention relates to inflators for devices such as protective passive restraints or "air bags" used in motor vehicles, escape slide chutes, life rafts, and the like. More particularly, the present invention relates to gas-generating compositions which are used in inflators.

Background Art

Many devices, such as protective passive restraints or "air bags" used in motor vehicles, escape slide chutes, life rafts, and the like, are normally stored in a deflated state and are inflated with gas at the time of need. Such devices are generally stored and used in close proximity to humans and, therefore must be designed with a high safety factor which is effective at all times.

Inflation is generally accomplished by means of a gas, such as air, nitrogen, carbon dioxide, helium, and the like which is stored under pressure and further pressurized and supplemented at the time of use by the addition of high temperature combustion gas products produced by the burning of a gas-generating composition. In some cases, the inflation gases are solely produced by gas-generating compositions.

The components of gas-generating compositions are incorporated into mechanical inflator devices in the form of powders, grains, pellets, or the like. A particular convenient manner to incorporate gas-generating

compositions into inflator devices is to form or mold the compositions into solid structures.

One concern with molded gas-generating components is that in order to be moldable they generally include binder components, some of which are suspected to produce hazardous combustion products. For example, polyvinyl chloride (PVC) is suspected of producing polychlorinated biphenyls (PCB's) when it is combusted at elevated temperatures.

The present invention is directed to moldable gas-generating compositions which do not include binder components which are suspected of producing harmful combustion products. More particularly, the present invention is directed to moldable gas-generating compositions which avoid the use of polyvinyl chloride (PVC) binders.

#### Disclosure of the Invention

It is accordingly an object of the present invention to provide gas-generating compositions which can be molded into solid structures.

Another object of the present invention is to provide gas-generating compositions which can be injection molded or extruded.

It is another object of the present invention to provide moldable gas-generating compositions which do not include binder components which produce harmful combustion products.

A further object of the present invention is to provide moldable gas-generating compositions which do not include polyvinyl chloride binder components.

A still further object of the present invention is to provide moldable gas-generating compositions which include hydroxyl terminated polybutadiene (HTPB) binder systems.

A still further object of the present invention is to provide moldable gas-generating compositions which can be quick cured.

5 A still further object of the present invention is to provide moldable gas-generating compositions which can be cured in approximately 7 minutes or less.

A yet further object of the present invention is to provide a method of molding gas-generating compositions.

10 A yet further object of the present invention is to provide a method of molding gas-generating compositions which include hydroxyl terminated polybutadiene (HTPB) binder systems which can be quick cured.

15 According to these and further objects of the present invention which will become apparent as the description thereof proceeds, the present invention provides a moldable gas-generating composition which includes:

an oxidizer component;  
20 a hydroxyl-terminated polybutadiene binder component;

a curing agent for curing the hydroxyl-terminated polybutadiene binder component; and

a cure catalyst for accelerating the curing rate of the hydroxyl-terminated polybutadiene component.

25 The present invention further provides a method of forming a molded gas-generating composition which comprises:

30 forming a moldable gas-generating composition which includes an oxidizer component, a hydroxyl-terminated polybutadiene binder component, a curing agent for curing the hydroxyl-terminated polybutadiene binder component, and a cure catalyst for accelerating the curing rate of the hydroxyl-terminated polybutadiene component;

35 shaping the moldable gas-generating composition; and curing the shaped gas-generating composition.

5 The present invention further provides an inflator for inflating emergency devices which inflator includes a gas-generating composition that consists essentially of an oxidizer component and a hydroxyl-terminated polybutadiene binder component.

Best Mode for Carrying out the Invention

10 The present invention is directed to gas-generating compositions which, upon ignition, rapidly generate large amounts of gaseous reaction products. The gas-generating compositions of the present invention are moldable. In this regard, they can be prepared so as to have a suitable viscosity for injection molding, extrusion, or the like. After molding the composition can be cured to form solid structures

15 In use, the gas-generating compositions are molded into solid shapes which are incorporated into mechanical inflator devices such as protective passive restraints or "air bags" used in motor vehicles, escape slide chutes, life rafts, or the like. The present gas-generating  
20 compositions can be used in conjunction with inflator devices which primarily rely upon stored pressurized gas, and combustible gas-generating compositions to supplement the pressure of the stored gas at the time of use. Alternatively, the present gas-generating compositions  
25 can be used as the primary source of gas used to inflate an inflation device.

30 When incorporated into mechanical inflator devices, the gas-generating compositions of the present invention can be ignited by a conventional initiator or ignitor. For example, when used in conjunction with protective passive restraints or "air bags" used in motor vehicles, electric squibs which are activated upon a sensed impact of the motor vehicle can be used to ignite the gas-generating compositions.

5 The gas-generating compositions of the present invention include an oxidizer component, a binder component which serves as a fuel, a curing agent for the binder component, and a cure catalyst. For purposes of gas generation, the essential components include the oxidizer component and the binder component. For purposes of molding and curing the gas-generating composition, the curing agent and cure catalyst can be considered essential components. However, it is to be understood that the cure catalyst is only necessary when one desired to quicken the curing rate of the compositions.

10 Suitable oxidizers which can be used in the gas-generating compositions include alkali metal chlorates, alkali metal perchlorates, and mixtures thereof. Examples of these oxidizers include sodium chlorate, potassium chlorate, lithium chlorate, sodium perchlorate, potassium perchlorate, and lithium perchlorate. Other oxidizers which can be use include alkaline earth metal perchlorates and ammonium perchlorate.

20 One oxidizer which has been found to be particularly useful for purposes of the present invention is potassium perchlorate.

25 The preferred binder component used in the gas-generating compositions is a hydroxyl-terminated polybutadiene (HTPB). This binder functions as both a binder and a fuel component in the composition. Hydroxyl-terminated polybutadiene has been found to be a desirable binder component since it does not produce hazardous compounds upon combustion.

30 A curing agent for the binder component is included in the gas-generating composition. The curing agent causes the binder component to cure during the molding process.

35 Preferable curing agents include isocyanates and diisocyanates, particularly di-polyfunctional



diisocyanates. Exemplary curing agents include hexamethylene diisocyanate, poly phenylmethylene isocyanate, isophorone diisocyanate, dimeryl diisocyanate, and the like.

5 In addition to the curing agent, a cure catalyst can be included in the gas-generating compositions. The cure catalyst accelerates the curing speed of the gas-generating compositions so that they can be quick cured after molding.

10 Suitable curing catalysts include triphenylbismuth, dibutyltin dilaurate, and similar catalysts which are known to aid in the curing of hydroxyl-terminated polybutadiene. The curing catalyst accelerates the curing of the gas-generating compositions so that they  
15 can be molded quickly.

In preferred embodiments the oxidizer component comprises about 83 to 95 weight percent of the gas-generating compositions, the binder component and the curing agent together comprise about 5 to 17 weight  
20 percent of the composition, and the cure catalyst comprises about 0.025 to 0.5 weight percent of the composition.

In more preferred embodiments the oxidizer component comprises about 85 to 90 weight percent of the gas-generating compositions, the binder component and the curing agent together comprise about 10 to 15 weight  
25 percent of the composition, and the cure catalyst comprises about 0.025 to 0.5 weight percent of the composition.

30 In even more preferred embodiments the oxidizer component comprises about 88 weight percent of the gas-generating compositions, the binder component and the curing agent together comprise about 11.8 to 11.9 weight percent of the composition, and the cure catalyst  
35 comprises about 0.1 to 0.2 weight percent of the composition.

The gas-generating compositions of the present invention have a cure rate of less than 7 minutes and more typically between about 3 to 5 minutes, over a temperature range of about 200 to 375°F. The curing rate of the gas-generating compositions of the present invention is dependent upon the curing temperature, as one would expect. The fastest curing times are obtained at higher curing temperatures. The upper limit of the curing temperature is just below the decomposition temperature of the composition. Therefore, for purposes of the present invention, the upper cure temperature should be limited to about 375°F.

In preferred embodiments, a composition which included about 88 weight percent of the oxidizer component, about 11.8 to 11.9 weight percent of the binder component, and about 0.1 to 0.2 weight percent of the cure catalyst was found to have a curing rate of between about 3 to 5 minutes at 350°F.

The gas-generating compositions of the present invention are prepared by mixing the individual components together. In preparing the composition, the binder component and oxidizer component can be premixed together. It is preferred to add the curing agent and cure catalyst to the binder component and oxidizer component just prior to molding or extruding the composition so that the composition does not begin to cure prematurely.

The components can be mixed together utilizing conventional mixers, blenders, mills, etc. which are known to be useful for mixing pyrotechnic compositions.

During a typical extrusion process, an extrudable mass of the composition is prepared by mixing the components together. The extrudable mass is then fed into an extruder, extruded and blocked as desired. Next the extrudable mass is extruded, cut free and cured.

The viscosity of the mixed composition can be adjusted as necessary by incorporating a removable solvent such as ethyl acetate, acetone, ethyl alcohol, or mixtures thereof. The necessary viscosity for extrusion (or injection molding) can easily be determined based upon the specifications of the processing equipment used.

In addition to the above-discussed components, other components such as conventional stabilizers, colorants, opacifiers, and the like can be included as desired. A preferred stabilizer used in the examples which follow includes Triphenylbismuth, Maleic Anhydride, and Magnesium Oxide used together in substantially equal proportions or about 2:1:1.

Features and characteristics of the present invention will be further understood from the following non-limiting examples which are included for exemplary purposes. In these examples and throughout the specification, percentages are given as weight percents unless otherwise indicated.

#### Example 1

In this example four gas-generating compositions having the following formulations were prepared:

TABLE 1

Component	Parts by Weight			
Hydroxyl-terminated Polybutadiene	15.63	10.85	9.61	2.54
Isophorone Diisocyanate	1.37	0.95	0.84	0.39
Potassium Perchlorate	83	88	89.5	94
Triphenylbismuth Maleic Anhydride Magnesium Oxide	0.2	0.2	0.2	0.2

The above formulations were mixed, mold cured for 3 minutes at 350°F and found to have the following properties.

TABLE 2

## Performance Properties

Viscosity (K <sub>p</sub> )	2.5	5.5	5.3	5.3
Burning rates @ 1000 psi in/sec	0.80	0.87- 0.90	~0.95	1.73
Sensitivity --Impact, friction, ESO and auto ignition	Low	Low	Low	Low

## Mechanical Properties

Stress (psi)	118	151	212	38
Strain (%)	26	18	8	6.4
Modulus (psi)	1550	1800	4584	1250

This data indicates that the formulations meet the requirements for air bag generator (inflator) applications.

Although the present invention has been described with reference to particular means, materials and embodiments, from the foregoing description, one skilled in the art can easily ascertain the essential characteristics of the present invention and various changes and modifications may be made to adapt the various uses and characteristics without departing from the spirit and scope of the present invention as described by the claims which follow.

CLAIMS

1. A moldable gas-generating composition which comprises:

an oxidizer component;

5 a hydroxyl-terminated polybutadiene binder component;

a curing agent for curing said hydroxyl-terminated polybutadiene binder component; and

a cure catalyst for accelerating the curing rate of said hydroxyl-terminated polybutadiene component.

2. A moldable gas-generating composition according to claim 1, wherein said oxidizer component is selected from the group consisting of alkali metal chlorates, alkali metal perchlorates, alkaline earth metal chlorates, alkaline earth metal perchlorates, ammonium perchlorate, and mixtures thereof.

3. A moldable gas-generating composition according to claim 1, wherein said curing agent is selected from the group consisting of isocyanates, diisocyanates, and mixtures thereof.

4. A moldable gas-generating composition according to claim 1, wherein said cure catalyst is selected from the group consisting of triphenylbismuth, dibutyltin dilaurate, and mixtures thereof.

5. A moldable gas-generating composition according to claim 1, wherein said oxidizer component comprises about 85 to 95 weight percent of said gas-generating composition, said hydroxyl-terminated polybutadiene binder component and the curing agent together comprise about 5 to 15 weight percent of said gas-generating composition, and said cure catalyst comprises about 0.025

to 0.5 weight percent of said gas-generating composition.

5 6. A moldable gas-generating composition according to claim 5, wherein said oxidizer component comprises about 85 to 90 weight percent of said gas-generating composition, said hydroxyl-terminated polybutadiene binder component and the curing agent together comprise about 10 to 15 weight percent of said gas-generating composition, and said cure catalyst comprises about 0.025 to 0.5 weight percent of said gas-generating composition.

5 7. A moldable gas-generating composition according to claim 6, wherein said oxidizer component comprises about 88 weight percent of said gas-generating composition, said hydroxyl-terminated polybutadiene binder component and the curing agent together comprise about 11.8 to 11.9 weight percent of said gas-generating composition, and said cure catalyst comprises about 0.01 to 0.2 weight percent of said gas-generating composition.

8. A moldable gas-generating composition according to claim 1, which further includes a removable solvent.

9. A method of forming a molded gas-generating composition which comprises:

5 forming a moldable gas-generating composition which includes an oxidizer component, a hydroxyl-terminated polybutadiene binder component, a curing agent for curing said hydroxyl-terminated polybutadiene binder component, and a cure catalyst for accelerating the curing rate of said hydroxyl-terminated polybutadiene component;  
10 shaping said moldable gas-generating composition;  
and  
curing said shaped gas-generating composition.

10. A method of forming a molded gas-generating composition according to claim 9, wherein said gas-generating composition has a cure rate of 5 minutes or less at a temperature of 200 to 375°F.

11. A method of forming a molded gas-generating composition according to claim 9, wherein said shaping comprises injection molding or extruding said gas-generating composition.

12. A method of forming a molded gas-generating composition according to claim 9, wherein said gas-generating composition further includes a removable solvent.

13. A method of forming a molded gas-generating composition according to claim 9, wherein said moldable gas-generating composition comprises about 85 to 95 weight percent of said gas-generating composition, said hydroxyl-terminated polybutadiene binder component and the curing agent together comprise about 5 to 15 weight percent of said gas-generating composition, and said cure catalyst comprises about 0.025 to 0.5 weight percent of said gas-generating composition.

14. A method of forming a molded gas-generating composition according to claim 13, wherein said moldable gas-generating composition comprises about 85 to 90 weight percent of said gas-generating composition, said hydroxyl-terminated polybutadiene binder component and the curing agent together comprise about 10 to 15 weight percent of said gas-generating composition, and said cure catalyst comprises about 0.025 to 0.5 weight percent of said gas-generating composition.

15. A method of forming a molded gas-generating composition according to claim 14, wherein said moldable gas-generating composition comprises about 88 weight percent of said gas-generating composition, said hydroxyl-terminated polybutadiene binder component and the curing agent together comprise about 11.8 to 11.9 weight percent of said gas-generating composition, and said cure catalyst comprises about 0.01 to 0.2 weight percent of said gas-generating composition.

16. A method of forming a molded gas-generating composition according to claim 15, wherein said gas-generating composition has a cure time of about 3 minutes at a temperature of about 350°F.

17. In an inflator for inflating emergency devices which inflator includes a gas-generating composition, the improvement wherein said gas-generating composition consists essentially of an oxidizer component and a hydroxyl-terminated polybutadiene binder component.



## INTERNATIONAL SEARCH REPORT

International application N. —  
PCT/US96/13827**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(6) : C06B 45/10

US CL : 149/19.4, 19.9

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 149/19.4, 19.9

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US, A, 3692,495 (SCHNEITER et al.) 19 September 1972, col. 1, lines 45-50 and col. 4, lines 1-5.	1-17
Y, P	US, A, 5,486,248 23 (TAYLOR) 23 January 1996, col. 3-4, col. 4, lines 22-24, col. 9, lines 12-24.	1-17
Y	US, A, 4,925,909 (KUBOTA ET AL.) 15 May 1990, col. 3, lines 45-54, col. 9, lines 15-30 (Example 8).	1-17
Y	US, A, 4,184,031 (GRAHAM ET AL.) 15 January 1980, col. 3, lines 40-52.	1-17
Y	US, A, 4,670,068 (CHI) 02 June 1987, col. 2, lines 16-23.	1-17

☒ Further documents are listed in the continuation of Box C.
 ☐ See patent family annex.

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## INTERNATIONAL SEARCH REPORT

International application N .  
PCT/US96/13827

## C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim N .
Y	US, A, 4,803,019 (GRAHAM ET AL.) 07 February 1989, col. 10, lines 12-22.	1-17
Y	US, A, 4,944,815 (CONSAGA) 31 July 1990, col. 3, lines 59-62 and col. 4, lines 23-27.	1-17
A, P	US, A, 5,472,532 (WALLACE II) 05 December 1995.	1-17
A, P	US, A, 5,474,625 (DUONG ET AL.) 12 December 1995.	1-17